Saturn Helium Bottles

Contract NAS 8-11555

Report on Problems Associated with Heat Treatment

ER 13796

February 1965

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I. INTRODUCTION

During the course of fabricating helium bottles for NASA-MSFC under Contract NAS 8-11555, two problems have been encountered with regard to heat treatment. The first indication of a problem came on 7-13-64 when unit S/N 0018, the fourth in the first lot of five, experienced a volumetric increase of 443 cubic inches after proof test. The specifications allow no more than 225 cubic inches expansion. Nothing unusual in either the fabrication or testing of this unit was discernible. The fifth unit in this lot expanded only 31 cubic inches. In the next lot of five units, the first, S/N 0022, expanded 962 cubic inches, the second and third were normal and the fourth and fifth, S/N 0024 and 0014 expanded 948 and 403 cubic inches respectively. This last unit had been tested on 9-16-64 and had been heat treated in the latter part of June.

At this point in time it was decided that a complete survey of the Vendor's furnace was in order and that the third lot of five units would not be heat treated until it was certain we could obtain the desired heat treatment. The survey of the furnace revealed a heat leak which resulted in a localized area, about two feet in length near the furnace hearth, which was running about 40-50°F colder than the rest of the furnace. The cold spot was caused by a depressed area in the hearth which allowed an air passage to form which undoubtedly became progressively worse with use. It was ultimately found that a small piece of metal had lodged in the solenoid valve controlling the pilot in one combustion chamber. This prevented complete shutoff and the metal support for the hearth had been subjected to localized heating causing it to sag. Repairs were made and a resurvey after the repairs showed that the furnace was operating properly. While the surveys and repairs were being made, hardness measurements on the bottles showed that there was a marked difference between the overexpanded units and the others. It was felt, at this time, that the cold spot was a significant factor contributing to underheating of these units and that the hardness tests could distinguish a good unit from a bad one. It was decided, therefore, to start heat treating the third lot of five units.

The first of these units, S/N 0013, was heat treated in November 1964 and the hardness survey showed a marked increase over the units which had expanded. Confidence in the Vendor's equipment and in the basic process had been regained and orders to proceed were issued. The Vendor requested, and was given permission, to charge units into a hot furnace. The Martin Company could not see anything detrimental in this procedure and hoped to make up some lost time. The next four units were heat treated in November 1964 and all hardness surveys indicated good units. During December 1964, the mechanical property test results became available and the samples from three of the last four units, S/N 0023, 0025, 0026, had one or more elongation value below the minimum acceptable. The values obtained are shown in Table I.

TABLE I

Heat Treat Test Bar Properties

S/N	Spec. No.	UTS	YS	Elong.
0023	L1	81.2	75.2	6.0
0023	L4	81.3	76.0	5.5*
0023	T2	75.1	71.5	1.5**
0023	T3	75.7	71.9	1.5**
0025	L3	77.9	73.6	5.5*
0025	L5	78.6	73.2	6.5
0025	T2	75.4	71.6	4.0
0025	T4	76.8	72.5	4.0
0026	L1	76.3	69.4	5.5*
0026	L2	77.3	71.4	6.0
0026	T1	76.4	71.8	4.0
0026	T2	74.2	69.0	5.5

* Minimum required 6%

** Minimum required 2%

It was conjectured that overheating could account for the results obtained, and for the second time operations were suspended and an investigation was launched. Metallographic examination of the test bars confirmed the conjecture that they had been overheated. Metallographic examination of samples cut from the ends of the bottles, however, revealed that the bottles themselves had not been overheated. A bottle, available from another contract, was instrumented and utilized to obtain data on temperature vs. time starting both in a cold and hot furnace.

It is the purpose of this report to present these data and to analyze the problems encountered in the light of all available information.

[redacted]

II. DESCRIPTION OF THE HEATING PROCESS

A. The Furnace and Its Control

The furnace used is approximately 42 feet long by 2-1/2 feet square in the working zone. It is a gas-fired, air-circulating furnace with two independently controlled combustion chambers located below the floor of the furnace at mid length. The furnace has a door at either end but the bottles are always loaded through one door referred to as the front. The bottles are always located in the same position within the furnace because the thermocouple leads penetrate the furnace roof through fixed ports which must be aligned with holes in the bottle fixture. Since the fixture is approximately 20 feet long, the ends of the fixture are always approximately 11 feet from each furnace door. In addition, the bottles are always placed so that the end with the larger hole is closest to the rear door, in order to facilitate the internal quenching operation.

Each combustion chamber is controlled by a Leeds and Northrup proportional controller. The operation of the controller is basically as follows: The controller is set to a specific temperature called the set point or set temperature. The proportional band is next set at any desired value between 0 and 10% of the full scale temperature range. To be specific, suppose the set point is 950°F and the proportional band is 200°F (10% of 2000°F). In operation, the furnace control thermocouple (there is one for each zone controlled), signal is compared with the set point minus the proportional band. In the example, as long as the control couple reads less than 750°F, the gas flow control valve will be full open. Between 750 and 950°F the valve will be throttled in direct proportion to the difference between the set point and the control couple temperature.

In addition to the two control couples, there are twelve other couples strategically located within the work zone. The output of these couples is recorded on a strip chart. The heat input to the two zones of the furnace can be varied between zero and maximum, in an infinite variety of ways, depending upon how the operator adjusts the set point and proportional band in each zone.

B. Heating of the Bottles

As the temperature of the air in the furnace is raised, heat is transferred, by forced convection, to the outside surface of the bottle. Heat is then transferred through the wall of the bottle by conduction. Since the air flow around the outside of the bottle is symmetrical, there is no pressure differential across the length of the bottle and hence, no air flow through the bottle. The air inside the bottle is heated by natural convection, drawing heat from the inside bottle wall. A test bar placed inside the bottle will have its surface heated by this essentially still air and will transmit heat from its surface by conduction. A test bar outside the bottle will be heated in the same way except that it is in a forced convection environment. The various temperatures involved can be listed in order of decreasing magnitude as follows:

$$T_F \geq T_{TO} \geq T_{BO} \geq T_{BI} \geq T_{AI} \geq T_{TI}$$

where

T_F	is the furnace air temperature
T_{TO}	is the outside test bar surface temperature
T_{BO}	is the temperature of the outside surface of the bottle
T_{BI}	is the temperature of the inside surface of the bottle
T_{AI}	is the temperature of the air inside the bottle
T_{TI}	is the inside test bar surface temperature

A set of temperature data, illustrating the above, is shown in Fig. 1. For this run the furnace was brought up from room temperature with the bottle in the work zone. It can be seen that the outside test bar temperature leads the outside bottle surface temperature for about 150 minutes. The inside test bar temperature lags the outside bottle surface temperature for more than 200 minutes. A second set of data is presented in Fig. 2 for the case where the furnace was preheated to 945°F and then the load was inserted. From this figure, the same lead and lag trend can be seen but several important differences are noteworthy. First, the outside test bar temperature lead over the bottle is more pronounced. Second, the lag between the furnace air temperature and the outside test bar temperature is essentially zero after 30 minutes. Further study of Figs. 1 and 2 shows that in either case, the bottle surface temperature is about the same at 100 minutes. From this point on, the time it takes to bring the bottle wall into the desired range (935 $\pm$ 10°F) depends entirely on the furnace operator. In the run shown in Fig. 1, the controller set points were reduced at 125 minutes and increased at 175 minutes. As a result, the 30 minute hold period did not start until 436 minutes. In the run depicted in Fig. 2 the operator was urged to be less cautious. The controller was initially set at 960°F, at about 80 minutes it was reduced to 940°F and at about 130 minutes it was increased to 950°F. The result in this case was that the hold period started at 210 minutes.

Additional data is presented in Figs. 3 and 4 which shows how the temperature varies along the length of the bottle at various instants of time. A study of these figures will show that, in both cases, when the bottle reaches 900°F, the temperature is essentially uniform along its length. Note, however, in Fig. 4, the outside test bars are at 900°F after 46 minutes while the bottle is only about 800°F. In Fig. 3, for the cold furnace start, the test bars reach 900°F in about 126 minutes and the bottle is lagging only about 40°F.

FIG. 1 : TEMPERATURE-TIME HISTORY (COLD FURNACE START)

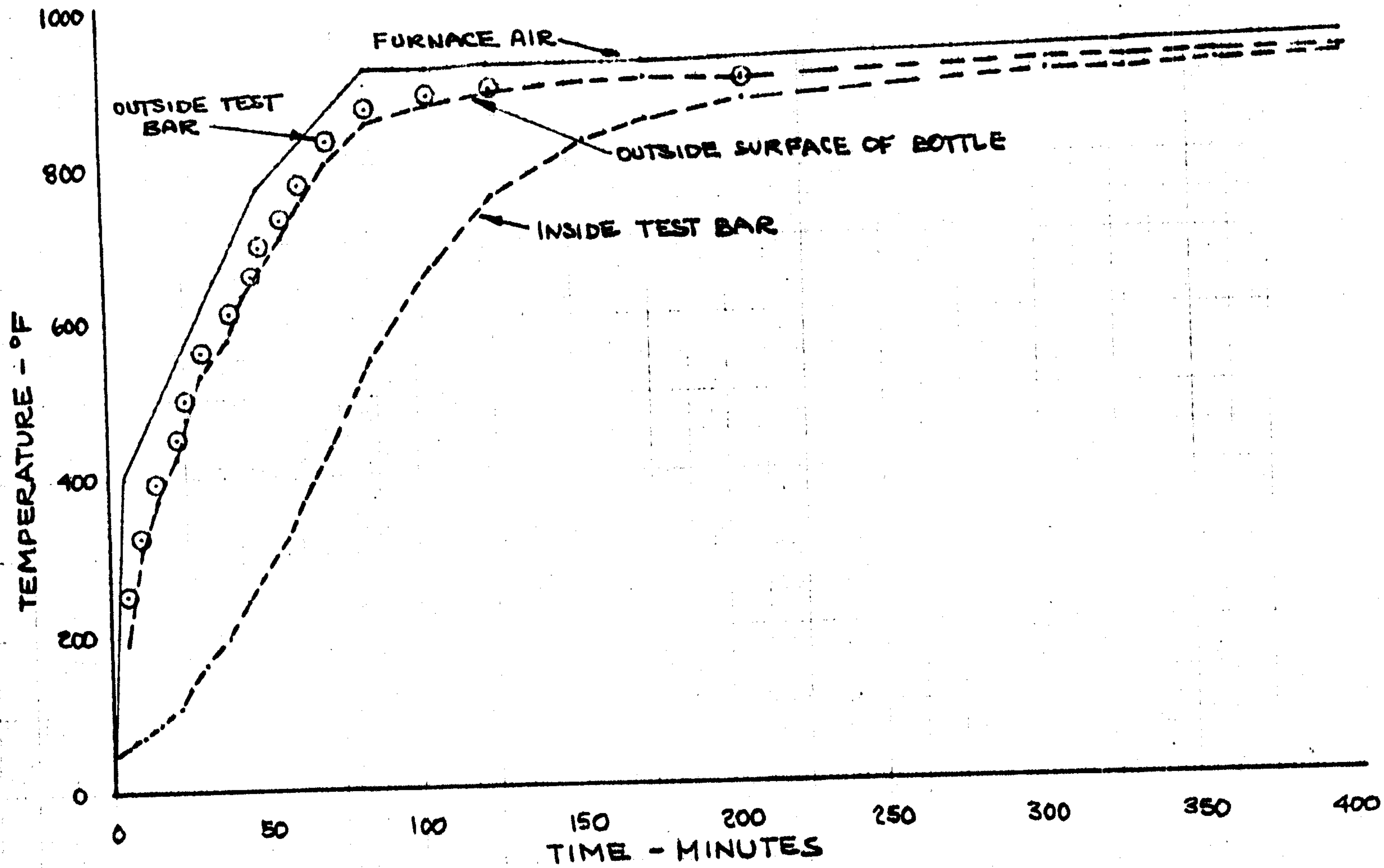


Fig. 2 :
TEMPERATURE-TIME HISTORY (HOT FURNACE START)

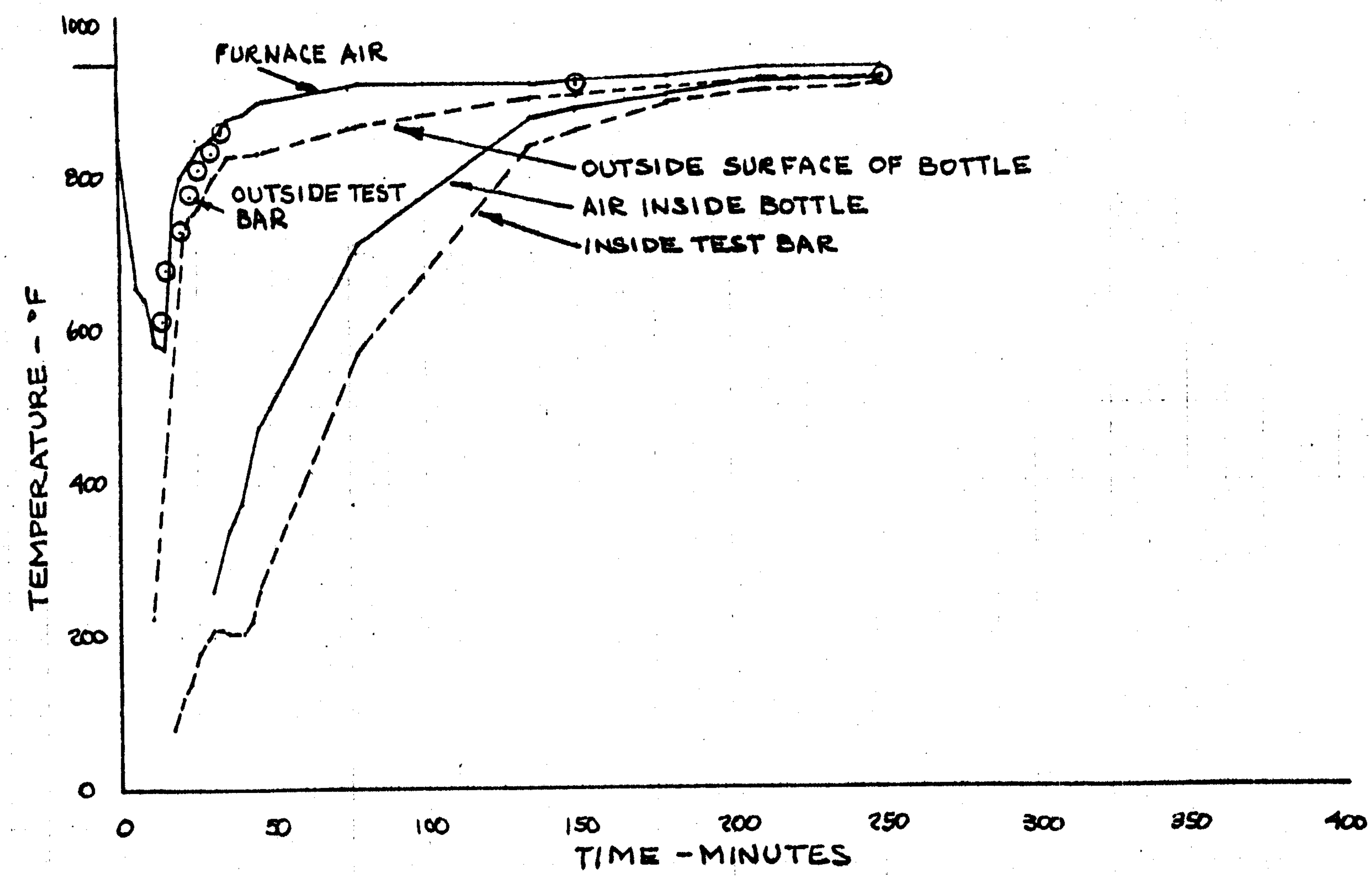


FIG. 3 : BOTTLE TEMPERATURE DISTRIBUTION
(STARTING WITH COLD FURNACE)

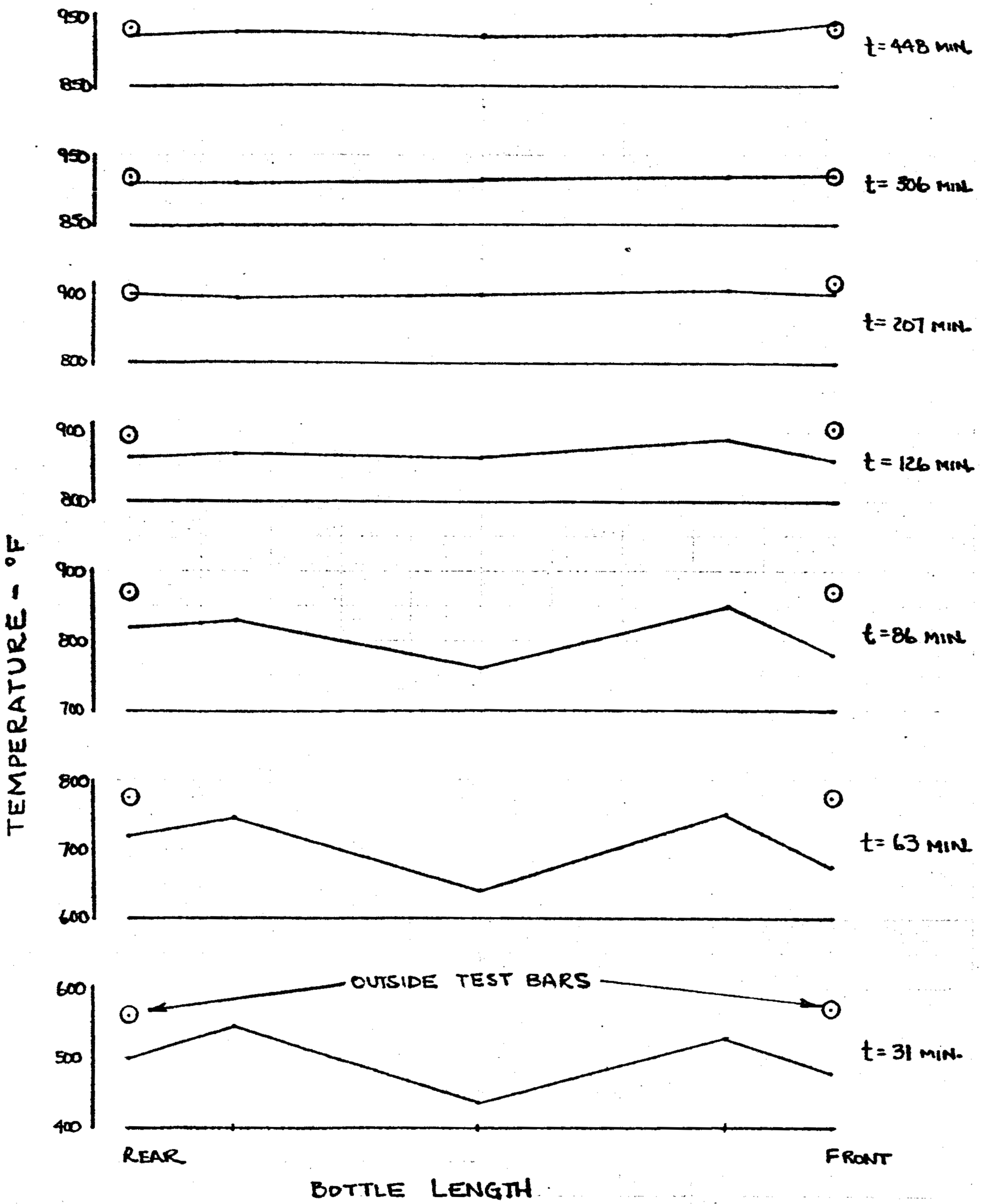
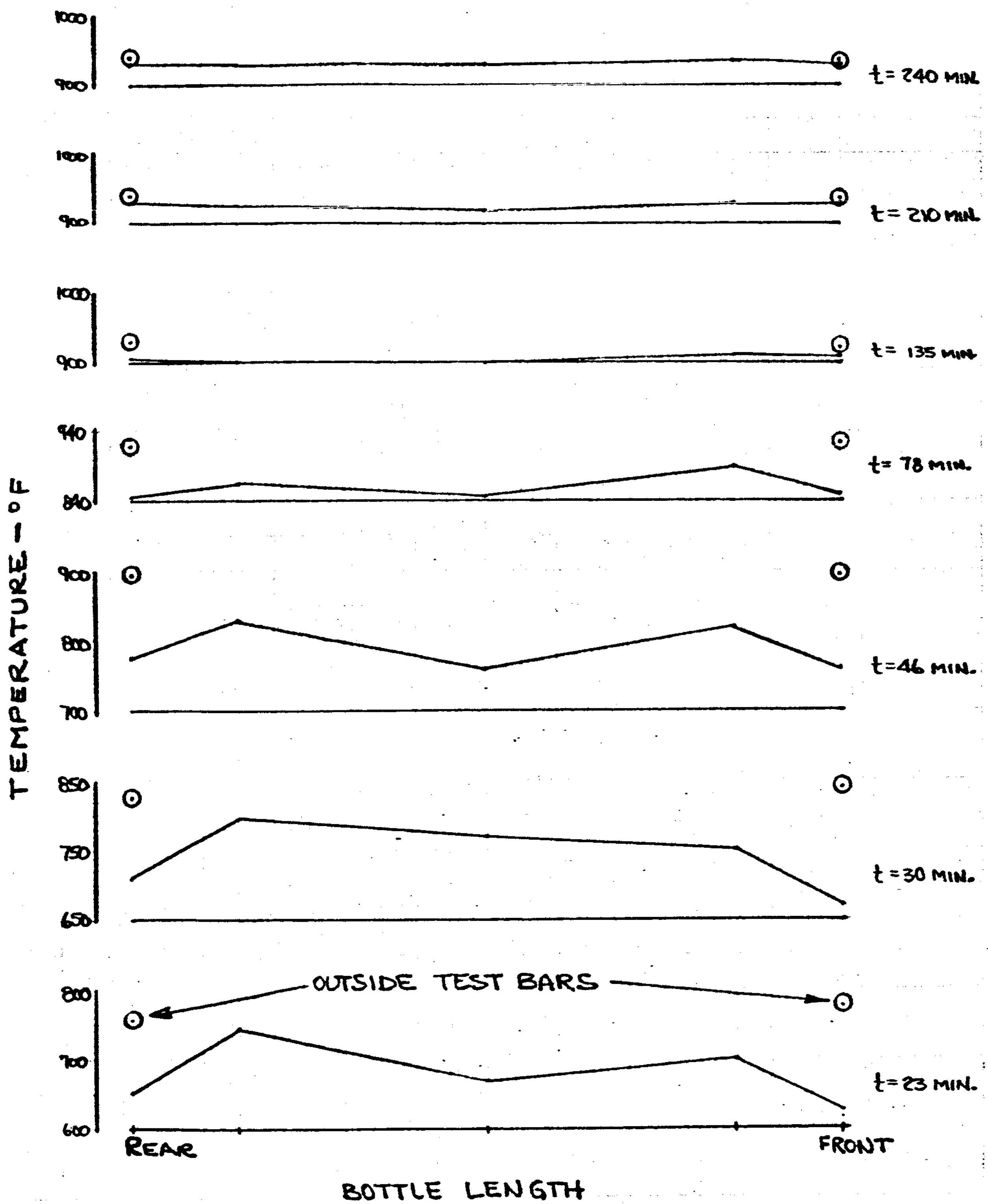


FIG. 4: BOTTLE TEMPERATURE DISTRIBUTION
(STARTING WITH HOT FURNACE)



III. THE UNDERHEAT PROBLEM

As stated in the Introduction, the underheating problem occurred in the first ten bottles produced under Contract NAS 8-11555. All of these bottles were heated starting with a cold furnace, using three thermocouples on the outside bottle surface and outside test bars only. Based upon subsequent hardness measurements it is known that four of the ten units (S/N 0015, 0016, 0017, 0020) were reasonably well heated while six of the units (S/N 0014, 0018, 0019, 0021, 0022, 0024) were not. It is also known that the test bars for all units were heat treated properly and that a furnace cold spot existed, probably, throughout this whole series. The data presented in Section II can be used to provide a logical explanation fitting all the known facts.

Figure 5 presents the temperature-time histories for S/N 0015, the best of the good units, and S/N 0022, the worst of the bad units. Figure 5 shows that up to about 150 minutes there was a significant difference in the rate of temperature rise. If Fig. 1 is compared with Fig. 5, it will be seen that the rate of rise on the test unit shown in Fig. 1 falls between the two curves shown in Fig. 5. It can be concluded that at any instant of time the lag between the outside bottle surface temperature and the inside surface temperature would be less for S/N 0015 than for S/N 0022. The effect of the cold spot in the furnace would probably increase this lag. It is further concluded, that during the hold period, even though both units had essentially the same temperature on the outside surface, the inside surface of S/N 0022 was lagging by as much as 50°F. The underheating of the six units would not have occurred if either the initial rate of heating had been higher or the total time for the cycle had been extended to 300-400 minutes.

The six suspect units are presently in the process of being reheat treated and additional controls have been imposed on the Vendor. These additions to the process will be discussed later in this report.

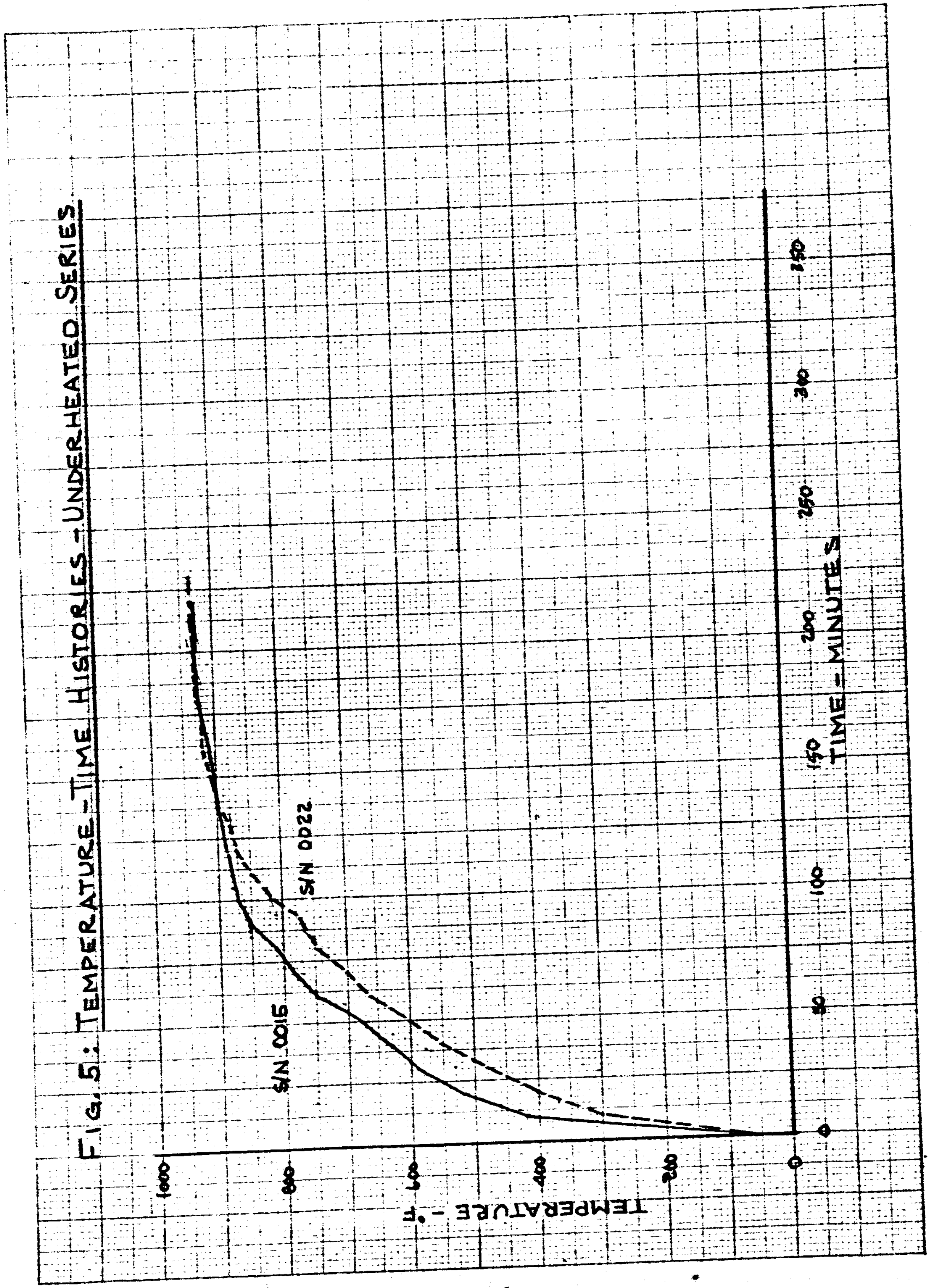


FIG. 5: TEMPERATURE - TIME HISTORIES - UNDERHEATED SERIES

IV. THE OVERHEAT PROBLEM

As stated in the Introduction, the last five units to be heat treated were charged into a hot furnace and subsequently three units (S/N 0023, 0025, 0026) were placed in a rejected status because the test bar elongation values were below the minimum specified (see Table I). During heat treatment these units had the same three external surface thermocouples as did the previous units. It was immediately suspected that the test bars had been overheated. The temperature charts did not indicate any overheating of the bottles. Each test bar that had a low elongation value was examined metallographically and it was verified that they had been overheated. At the same time, samples were removed from either end of all three bottles and these micrographs showed no evidence of overheating. All of these photomicrographs are presented herein as Appendix A. Figures 6 to 11 show the Temperature-Time Histories and Temperature Distributions for these three units. If these figures are compared with Figs. 2 and 4, it will be seen that these units were heated at a significantly higher rate than the test unit of Fig. 2. This means that, initially, the controller set points must have been higher than the test run, probably 960-970°F. Unfortunately, no permanent records exist as to the actual settings for the processing of these units. It is concluded, however, that during the early part of the cycle (0-100 minutes), the furnace air was as high as 960-970°F and from Fig. 2, the test bars reached the same range. The bottles, however, never exceeded 945°F. The evidence that the bottles were not overheated consists of the following:

1. No metallographic evidence in material from the bottle ends.

2. No temperatures above 945°F at three locations along the cylindrical portion of the bottle.

3. No significant difference between the temperatures at the ends of bottle as compared with the cylindrical portion for temperatures above 900°F (Fig. 4).

For completeness, the hardness data for these units is shown in Fig. 12.

Fig. 6: Temperature-Time History, S/N 0023, Initial Furn. Temp. 900°F

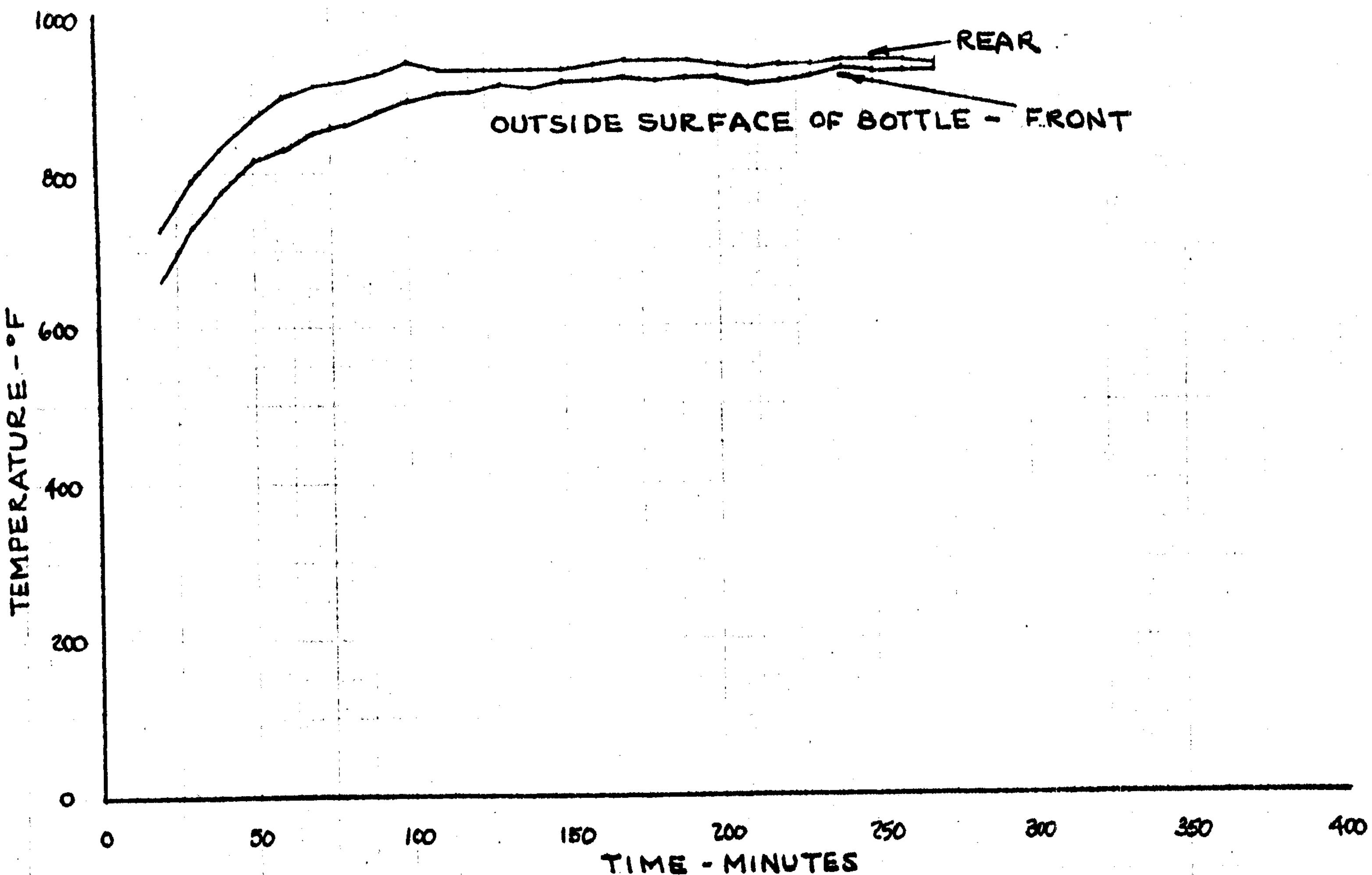


FIG. 7: BOTTLE TEMPERATURE DISTRIBUTION

S/N 0023 INITIAL FURN. TEMP. 900 °F

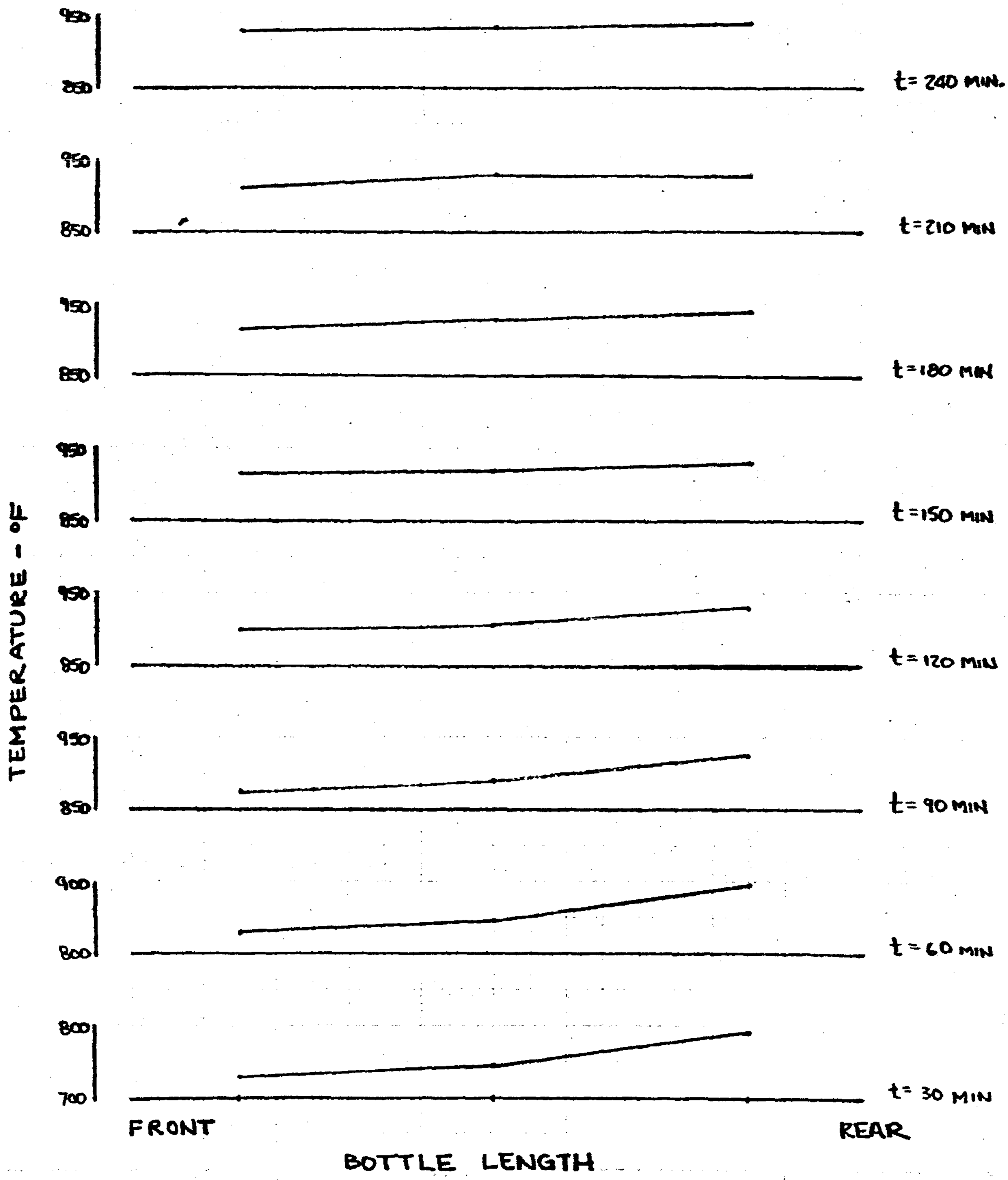


FIG. 8 : TEMPERATURE - TIME HISTORY, S/N 0025, INITIAL FURN. TEMP. 930°F

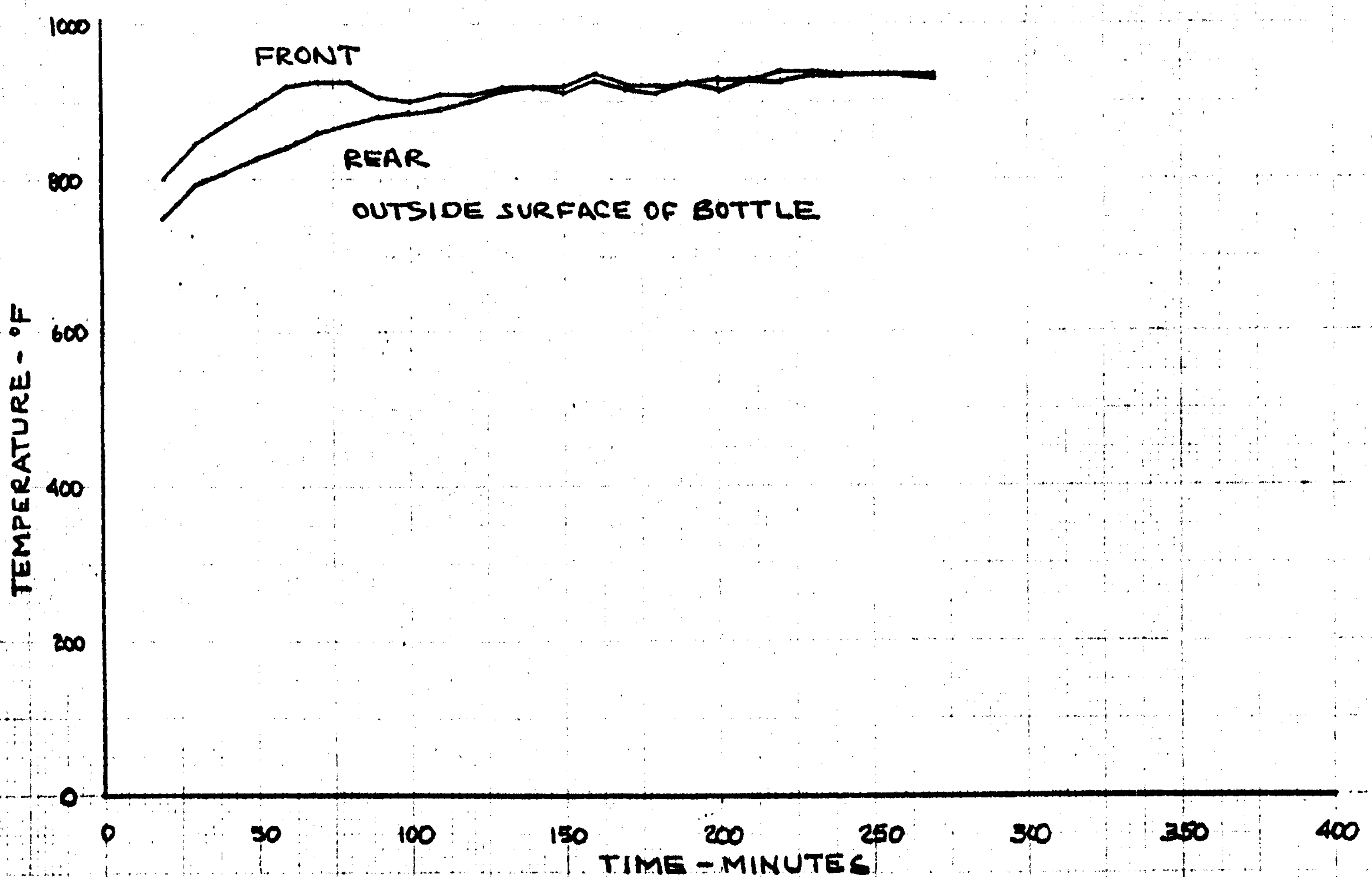


FIG. 9 : BOTTLE TEMPERATURE DISTRIBUTION

S/N 0025, INITIAL FURN. TEMP. 930 °F

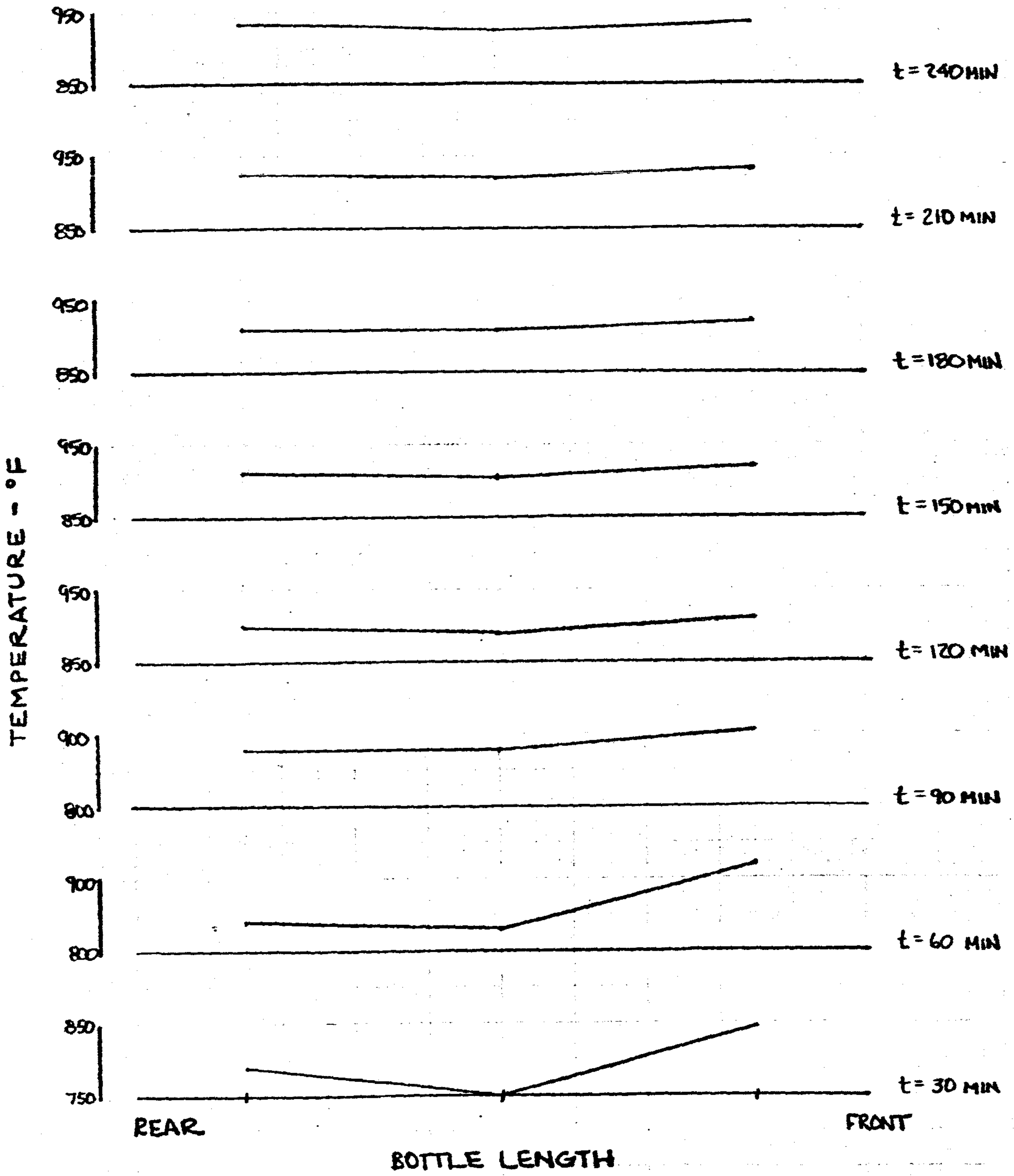


FIG. 10: TEMPERATURE - TIME HISTORY, S/N 0026, INITIAL FURN. TEMP. 930°F

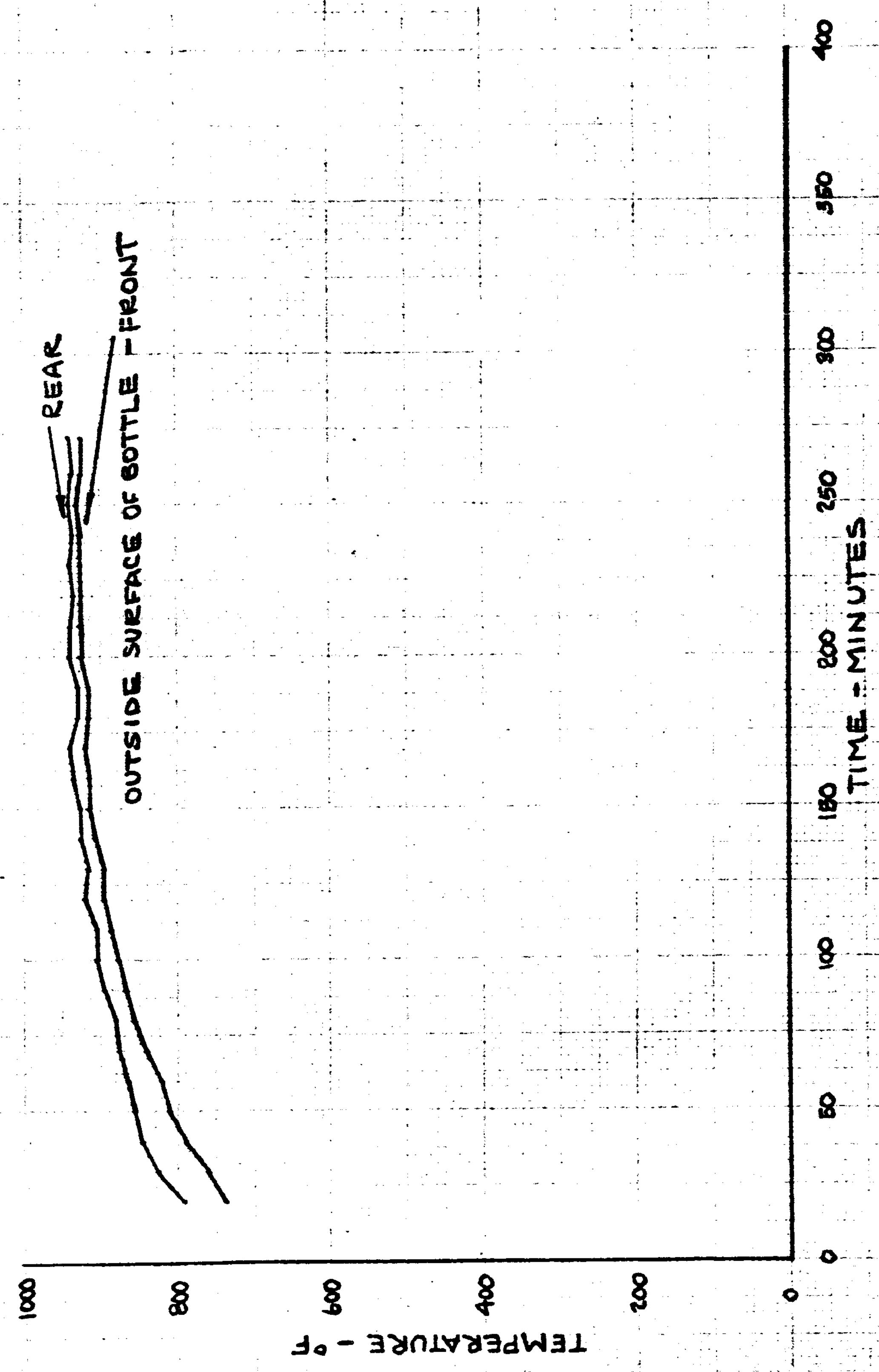
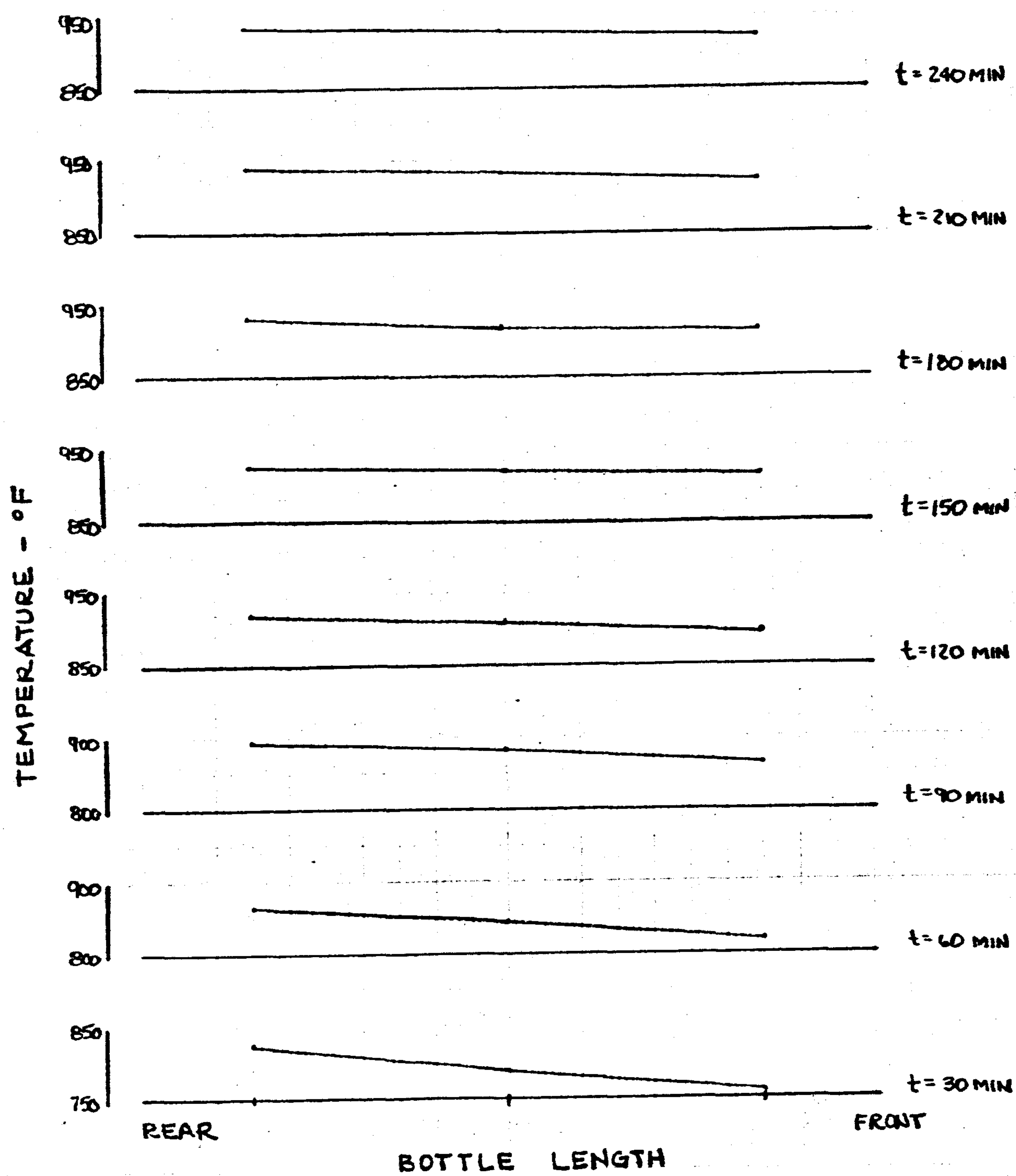


FIG. 11 : BOTTLE TEMPERATURE DISTRIBUTION

S/N 0026 INITIAL FURN. TEMP. 930°F



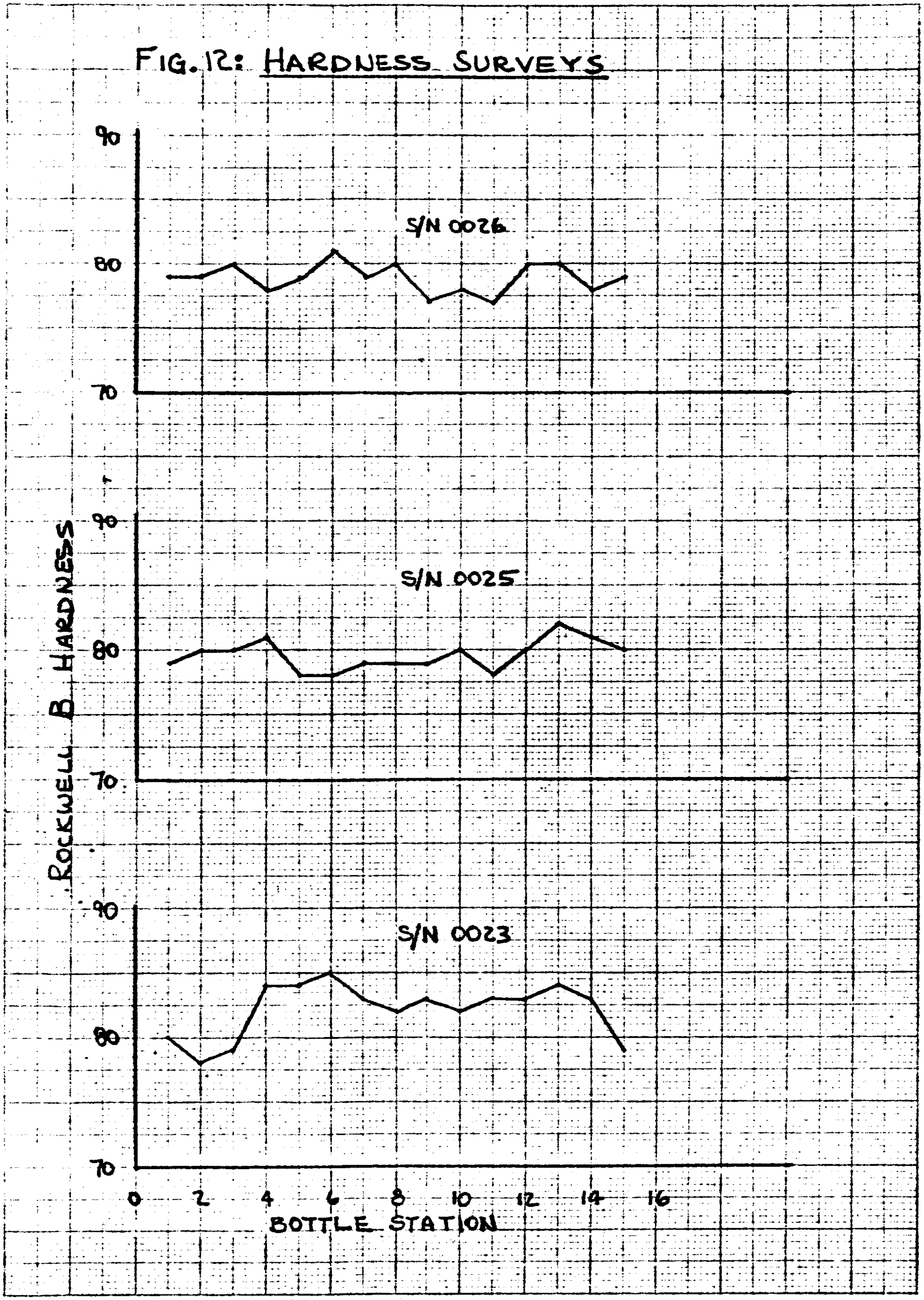


FIG. 12: HARDNESS SURVEYS
90
80
70
S/N 0026
S/N 0025
S/N 0023
ROCKWELL B HARDNESS
0
2
4
6
8
10
12
14
16
BOTTLE STATION

V. CONCLUSIONS

Based upon the data presented herein it is concluded that:

A. Furnace operator technique can result in underheating of bottles or overheating of test bars with the heat treat process as presently monitored and controlled.

B. Bottle S/N 0023, 0025, 0026 were not subjected to any overheating.

C. Additional controls are necessary to prevent a re-occurrence of these problems.

The following actions have been taken and made effective on all future bottles.

1. In addition to the three external bottle surface thermocouples, the temperature of the test bar at either end of the bottle will be recorded.

2. Four test bars with thermocouples attached will be placed inside the bottle, along its length.

3. Furnace controller set points will not exceed 950°F at any time.

4. The 30 minute hold cycle will not start until the external thermocouples are in the range 930-945°F and the internal thermocouples are in the range 925-945°F.

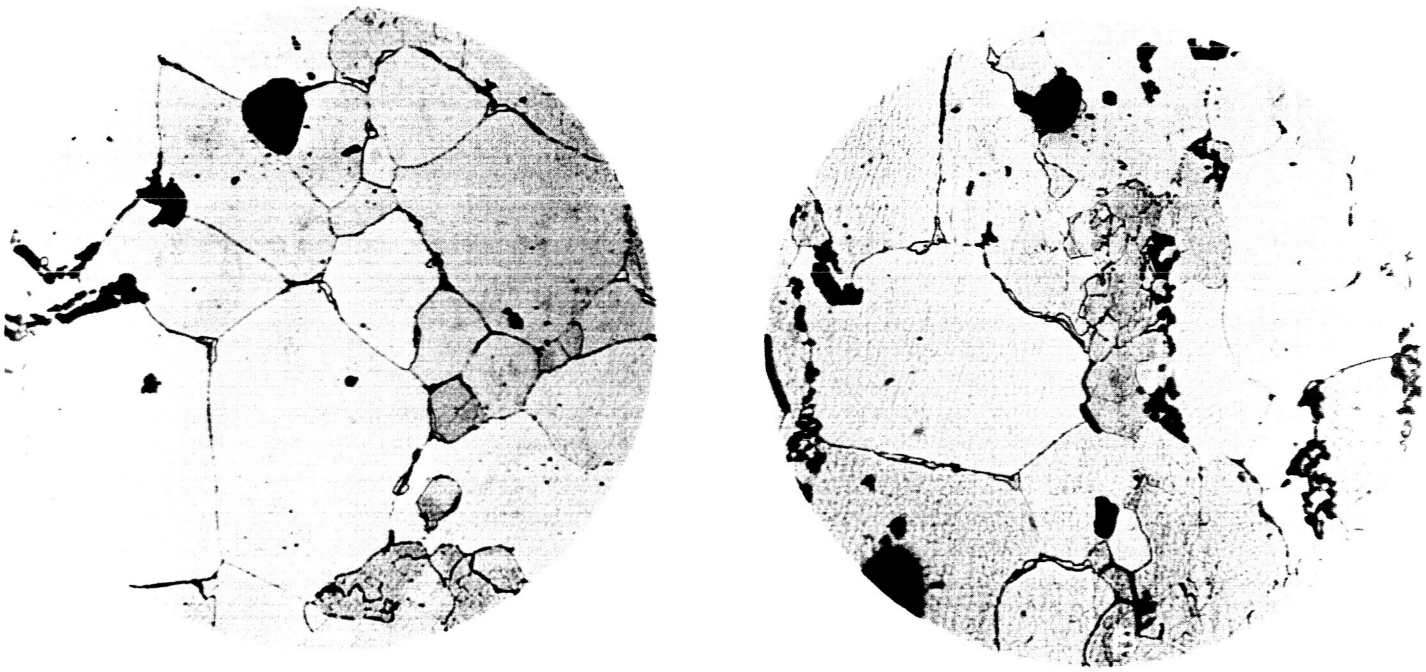
5. No bottle will be charged into the furnace unless the furnace temperature is below 350°F.

With the above restrictions, variations due to operator technique will be eliminated.

APPENDIX A

Photomicrographs for Bottles

S/N 0023, 0025, 0026

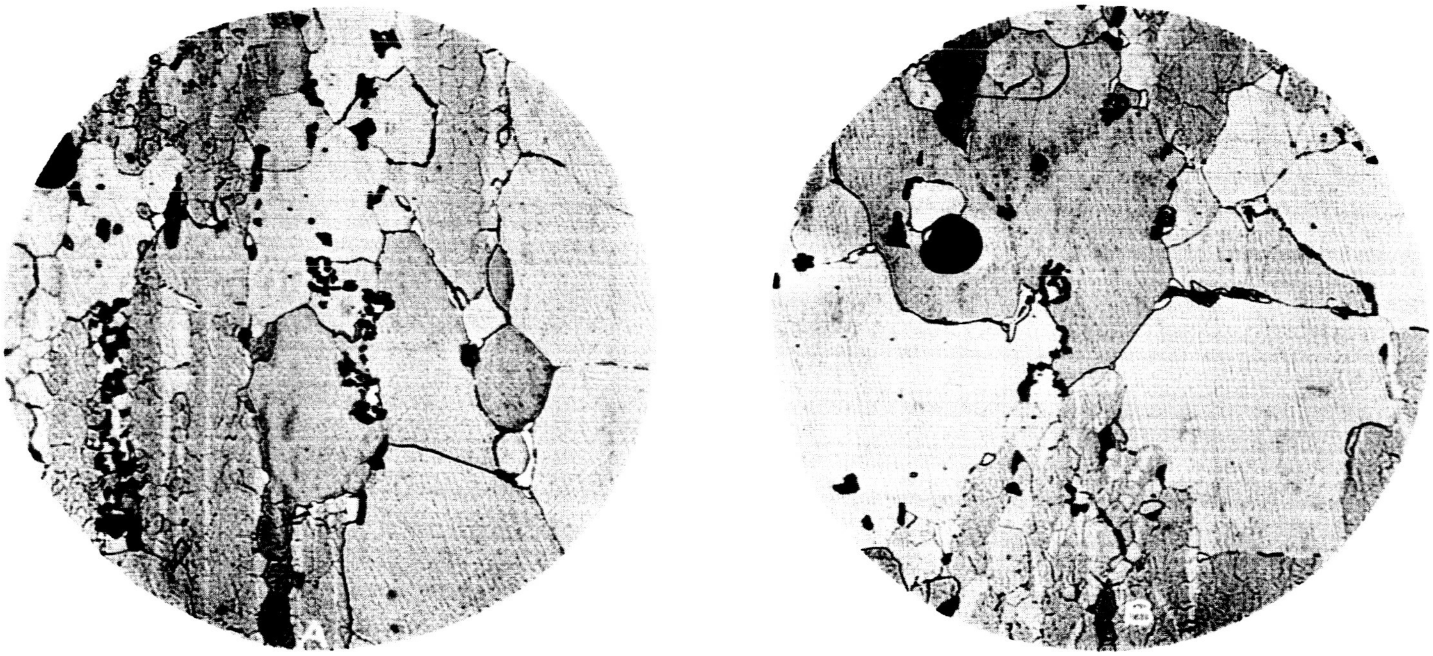


Saturn Helium Bottle

ML 11872C
Etchant:
Keller's

Metallographic structure of tensile test bar T2, S/N 0023, front position, showing extensive solid solution grain boundary melting and high temperature oxidation. UtS 75.1 ksi, TyS 71.5 ksi, Elong. 1.5%.

A-Longitudinal Plane
B-Long Transverse Plane
Magn. 400X



Saturn Helium Bottle

ML 11873C — Etchant: Keller's

Metallographic structure of tensile test bar L^4, S/N 0023, showing solid solution grain boundary melting, eutectic melting, and high temperature oxidation. UtS 81.3 ksi, TyS 76.0 ksi, Elong. 5.5%.

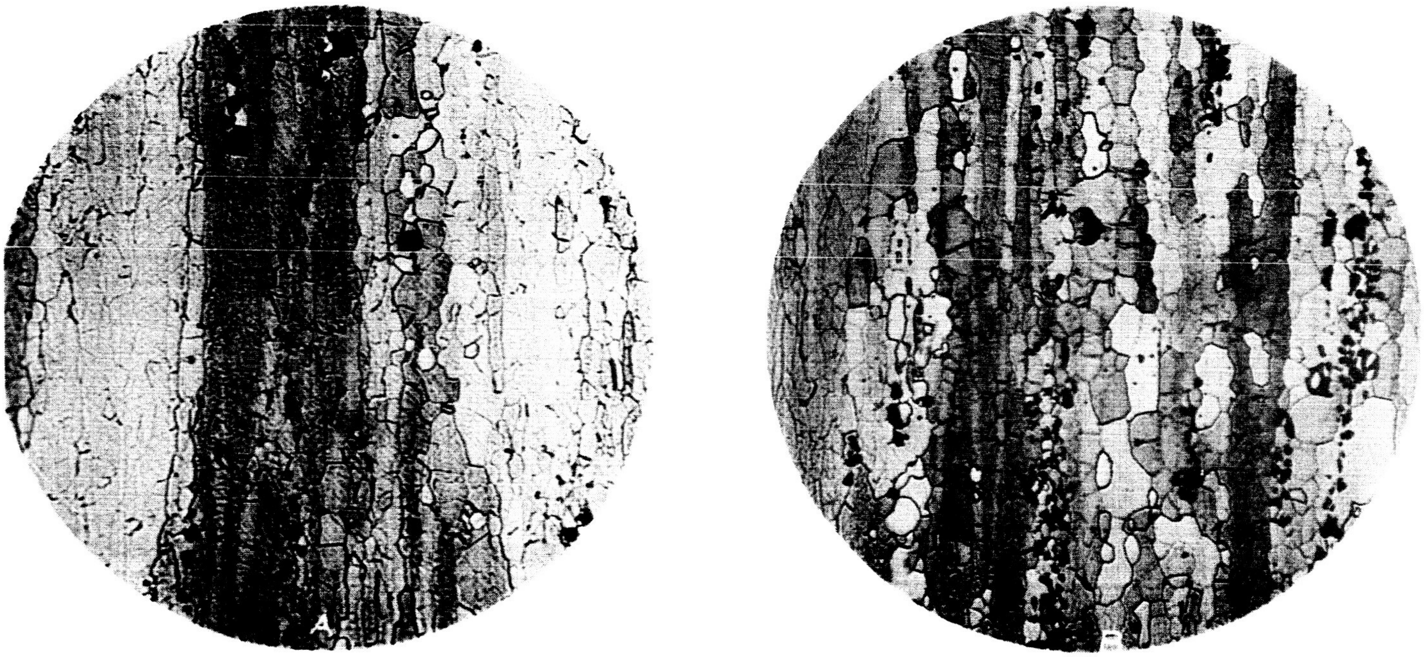
A-Longitudinal Plane
B-Long Transverse Plane
Magn. 400X

Saturn Helium Bottle

ML 11874C Etchant: Keller's

Metallographic structure of test section removed from front end position of bottle, S/N 0023, showing satisfactory heat treated condition for 2014-T6 alloy.

A-Longitudinal Plane
B-Long Transverse Plane
Magn. 400X

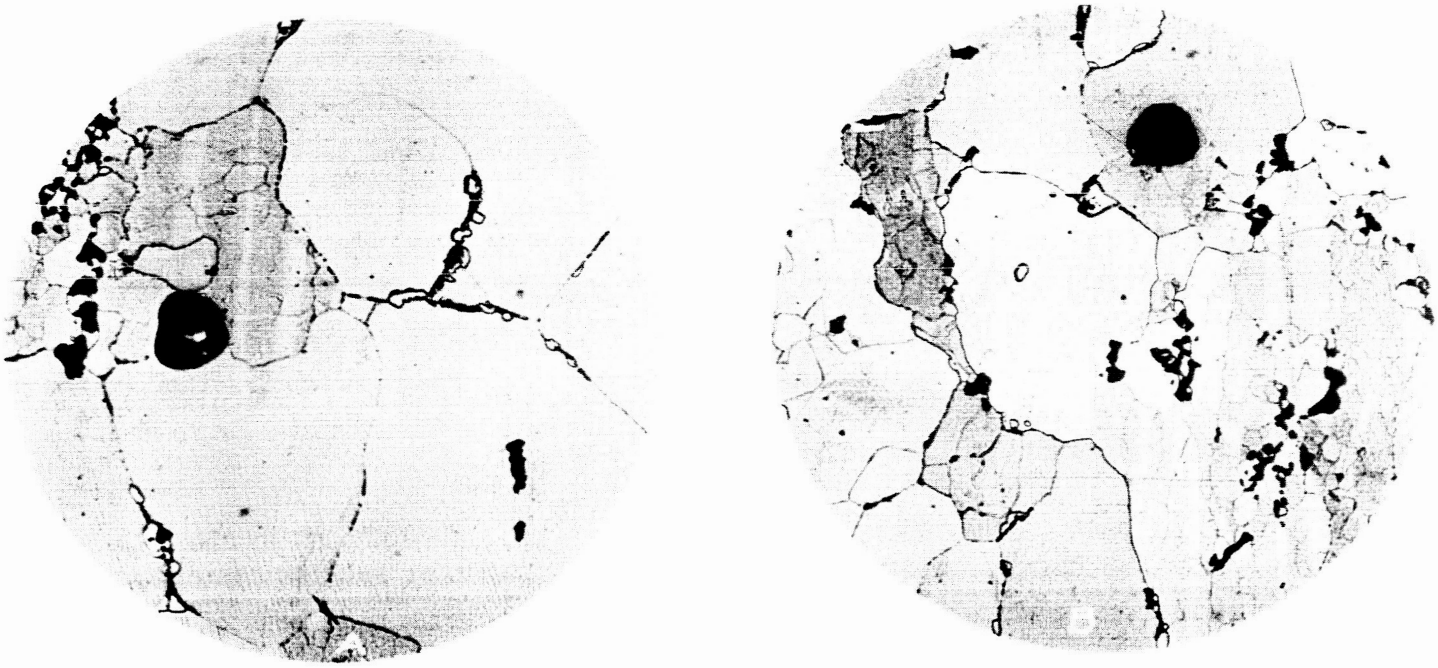


<u>Saturn Helium Bottle</u>

ML 11875C
Etchant:
Keller's

Metallographic structure of test section removed from back end position of bottle, S/N 0023, showing satisfactory heat treated condition for 2014-T6 alloy.

A-Longitudinal Plane
B-Long Transverse Plane
Magn. 400X

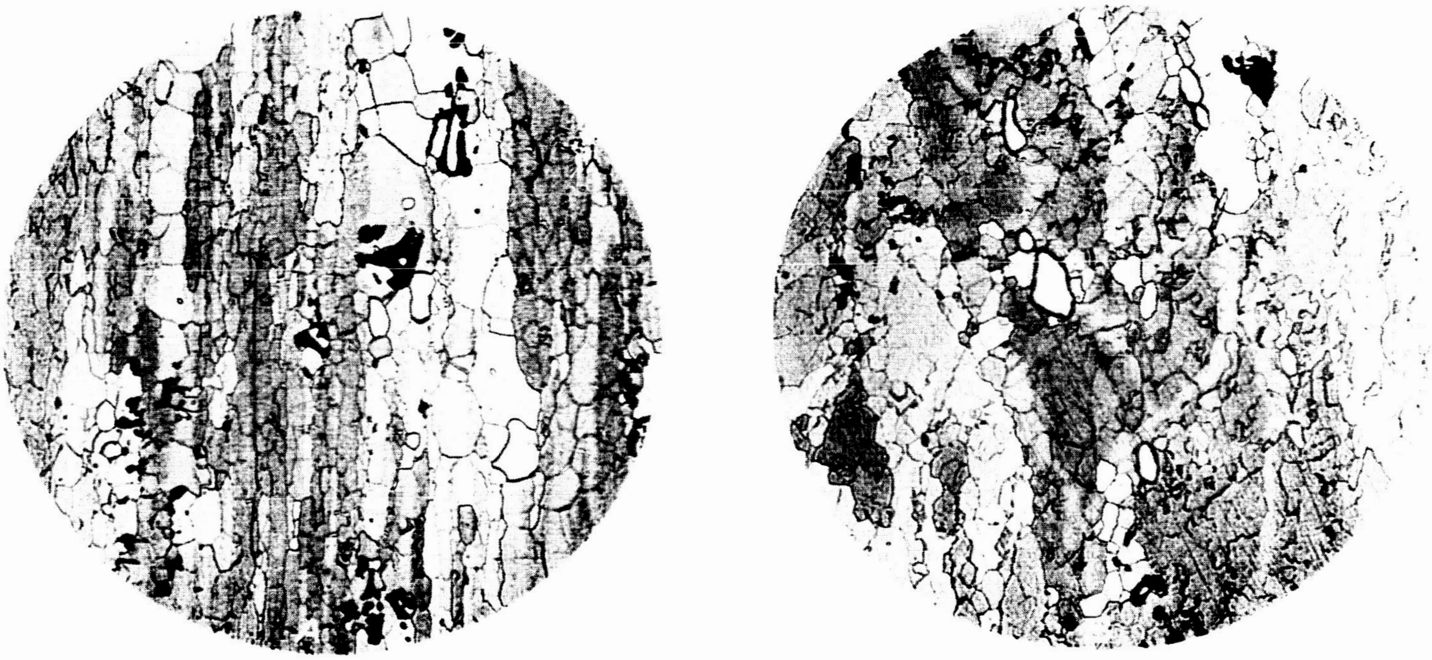


Saturn Helium Bottle

ML 11876C
Etchant:
Keller's

Metallographic structure of tensile test bar L3, S/N 0025, front end position showing extensive solid solution grain boundary melting and high temperature oxidation. UtS 77.9 ksi, TyS 73.6 ksi, Elong. 5.5%.

A-Longitudinal Plane
B-Long Transverse Plane
Magn. 400X

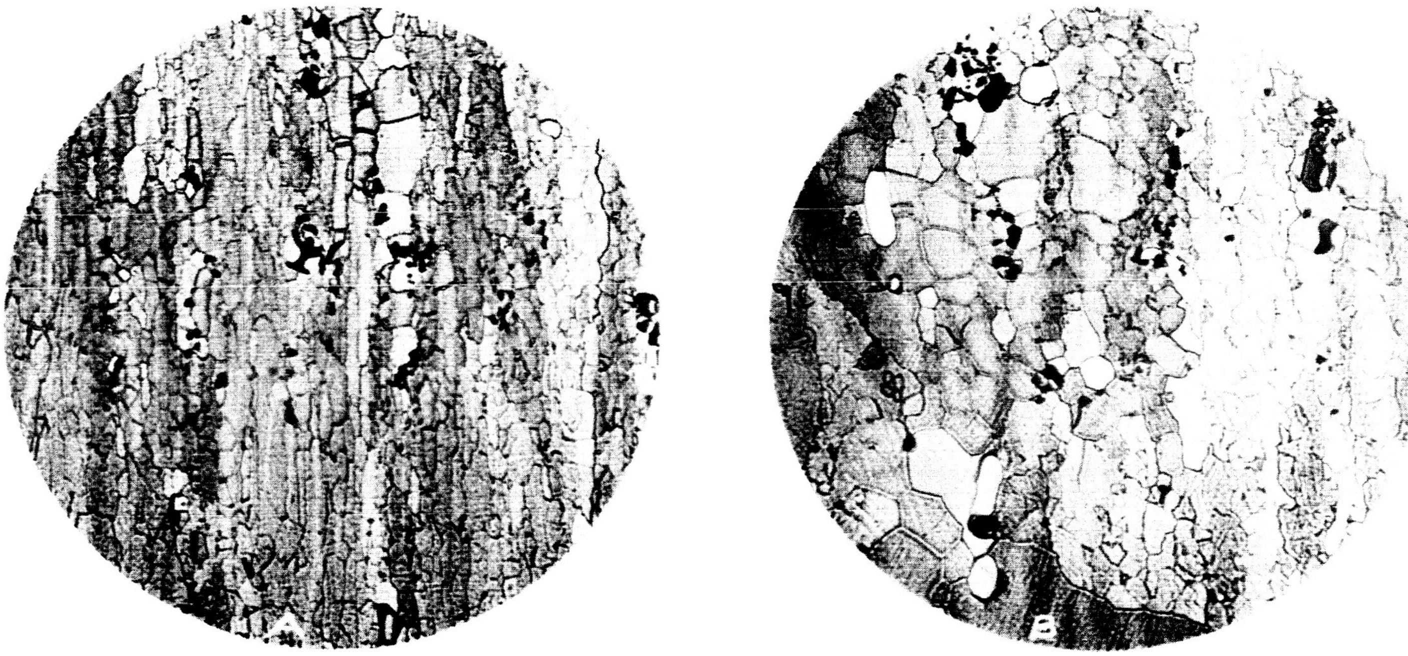


Saturn Helium Bottle

ML 11877C
Etchant:
Keller's

Metallographic structure of test section removed from front end position of bottle, S/N 0025, showing satisfactory heat treated condition for 2014-T6 alloy.

A-Longitudinal Plane
B-Long Transverse Plane
Magn. 400X

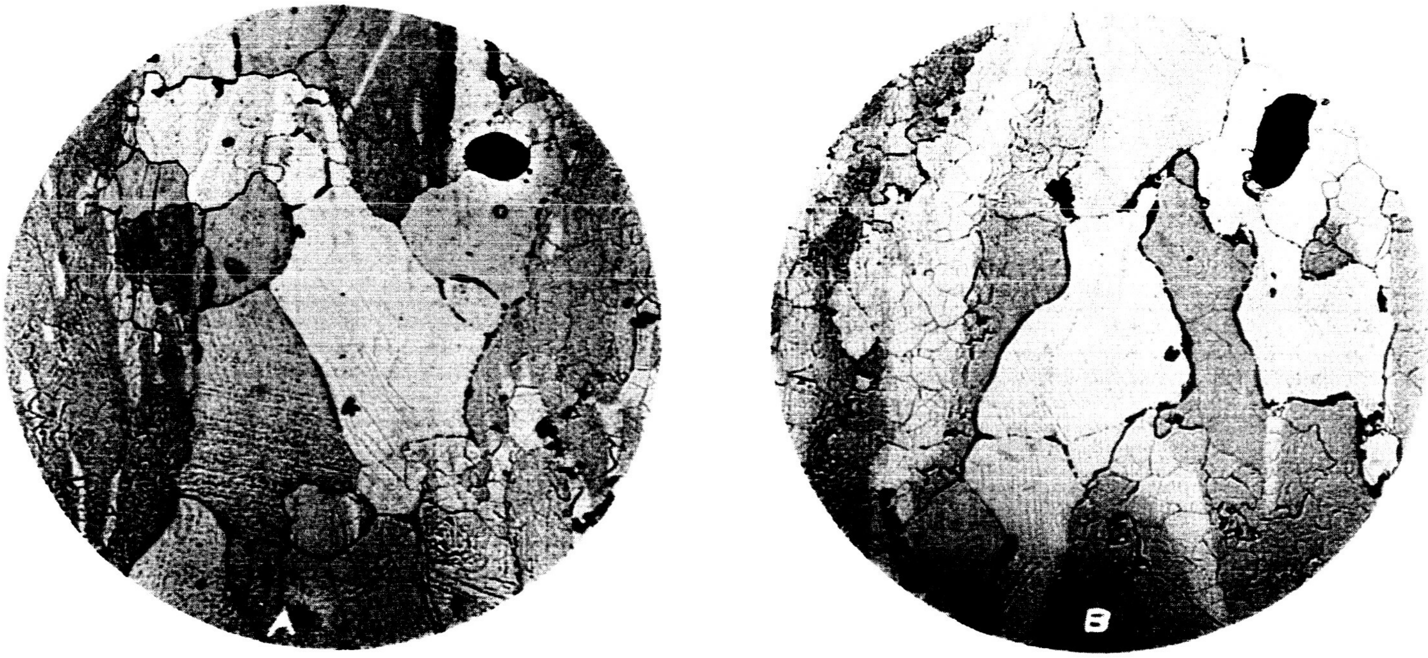


Saturn Helium Bottle

ML 11878C
Etchant:
Keller's

Metallographic structure of test section removed from back end position of bottle, S/N 0025, showing satisfactory heat treated condition for 2014-T6 alloy.

A-Longitudinal Plane
B-Long Transverse Plane
Magn. 400X



<u>Saturn Helium Bottle</u>

ML 11879C
Etchant:
Keller's

Metallographic structure of tensile test bar L1, S/N 0026, showing extensive solid solution grain boundary melting and high temperature oxidation. UtS 76.3 ksi, TyS 69.4 ksi, Elong. 5.5%.

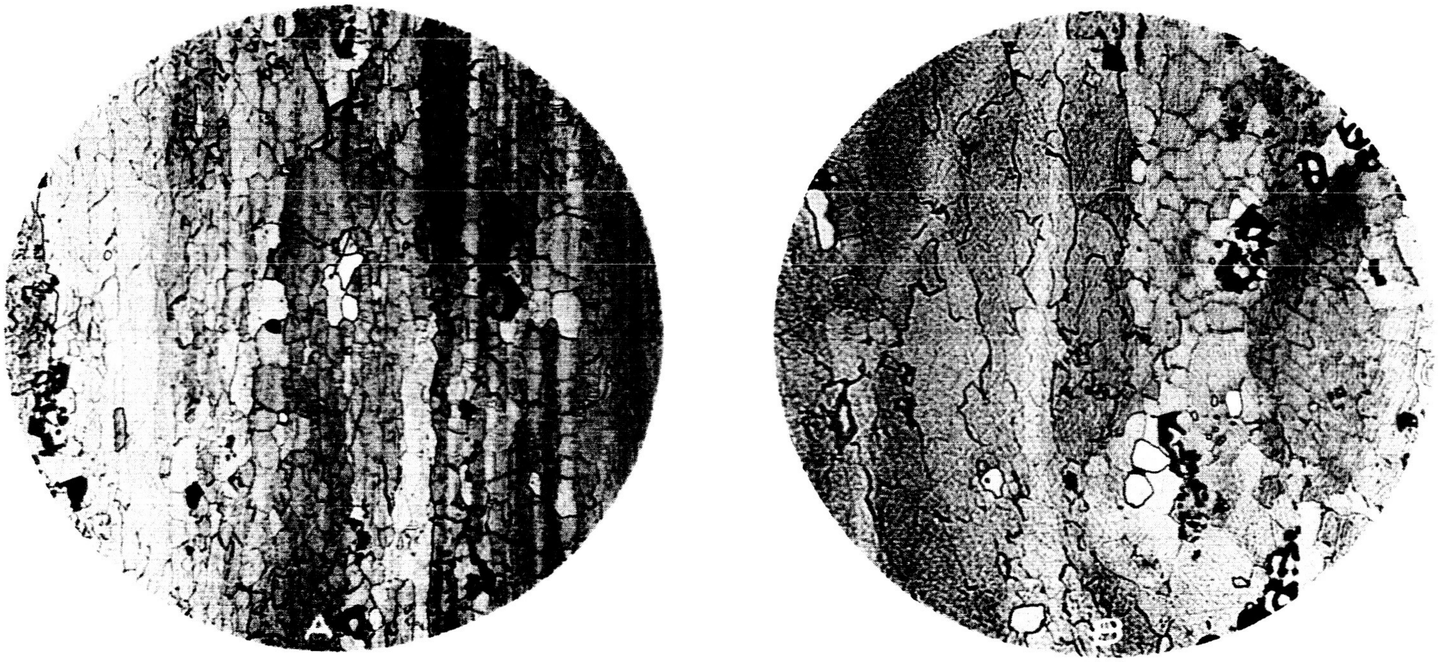
A-Longitudinal Plane
B-Long Transverse Plane
Magn. 400X

Saturn Helium Bottle

ML 11880C
Etchant:
Keller's

Metallographic structure of test section removed from front end position of bottle, S/N 0026, showing satisfactory heat treated condition for 2014-T6 alloy.

A-Longitudinal Plane
B-Long Transverse Plane
Magn. 400X



Saturn Helium Bottle

ML 11881C
Etchant:
Keller's

Metallographic structure of test section removed from back end position of bottle, S/N 0026, showing satisfactory heat treated condition for 2014-T6 alloy.

A-Longitudinal Plane
B-Long Transverse Plane
Magn. 400X

APPENDIX B

The Effect of Overheating on Saturn Helium Bottle Extrusion Material

In order to determine the possible effects of overheating on the extrusion material used in fabricating the bottles, a set of samples were prepared using material from extrusion S/N 0035. The mechanical properties, hardness, electrical conductivity and microstructure were determined in the as received condition, solution treated at 945°F, 950°F, 955°F, 960°F, 965°F and 970°F. All specimens heat treated were aged at 320°F for 20 hours. The data obtained is presented in Tables B1, B2, and B3.

TABLE B1

Mechanical Properties

Sol. Treat. Temp. °F	Yield (ksi) L	Yield (ksi) T	Ultimate L	Ultimate T	Elong. L	Elong. T
As received	68.7	63.2	75.4	69.0	9.0	5.0
945	72.1	57.5	77.6	72.8	9.0	4.5
950	67.6	70.6	74.0	72.8	9.0	4.5
955	72.7	70.8	79.1	73.5	8.0	4.5
960	70.2	70.2	74.5	71.9	4.0	2.0
965	75.1	71.4	81.0	74.6	6.5	2.0
970	70.6	70.6	74.8	74.3	3.0	2.0

TABLE B2

Rockwell B Hardness

Sol. Treat. Temp. °F	Hardness R_B
As received	81
945	84
950	85
955	87
960	87
965	87
970	87

TABLE B3

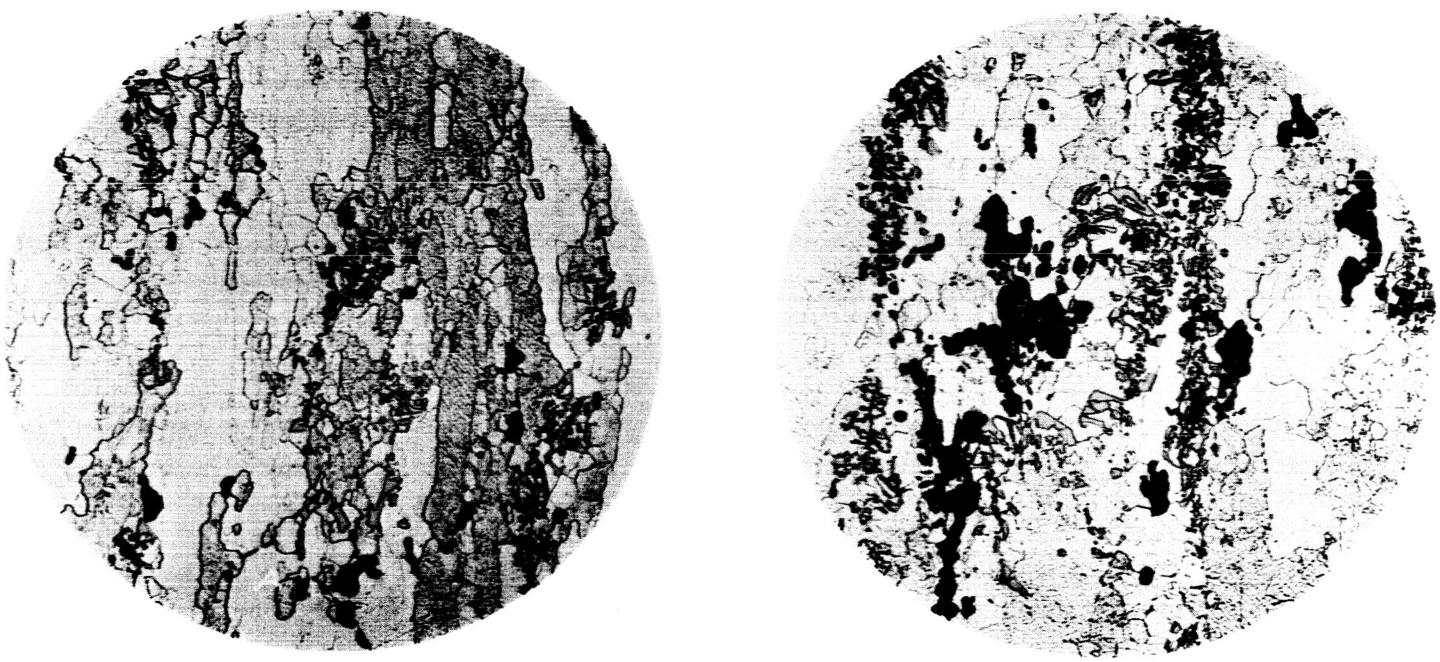
Electrical Conductivity

Sol. Treat. Temp. °F	Conductivity (% IACS) FM - 100*	FM - 100**	FM - 110
As received	38.65	38.99	38.00
945	37.45	37.95	37.25
950	37.30	37.75	37.25
955	36.85	37.50	37.00
960	36.00	36.25	35.95
965	35.25	35.50	35.40
970	35.55	35.99	34.95

* Engineering Lab Instrument

** Quality Lab Instrument

These data show that the elongation and conductivity experience a sharp decrease for a solution treating temperature above 955°F. A rather startling change in microstructure also is apparent between 955°F and 960°F.

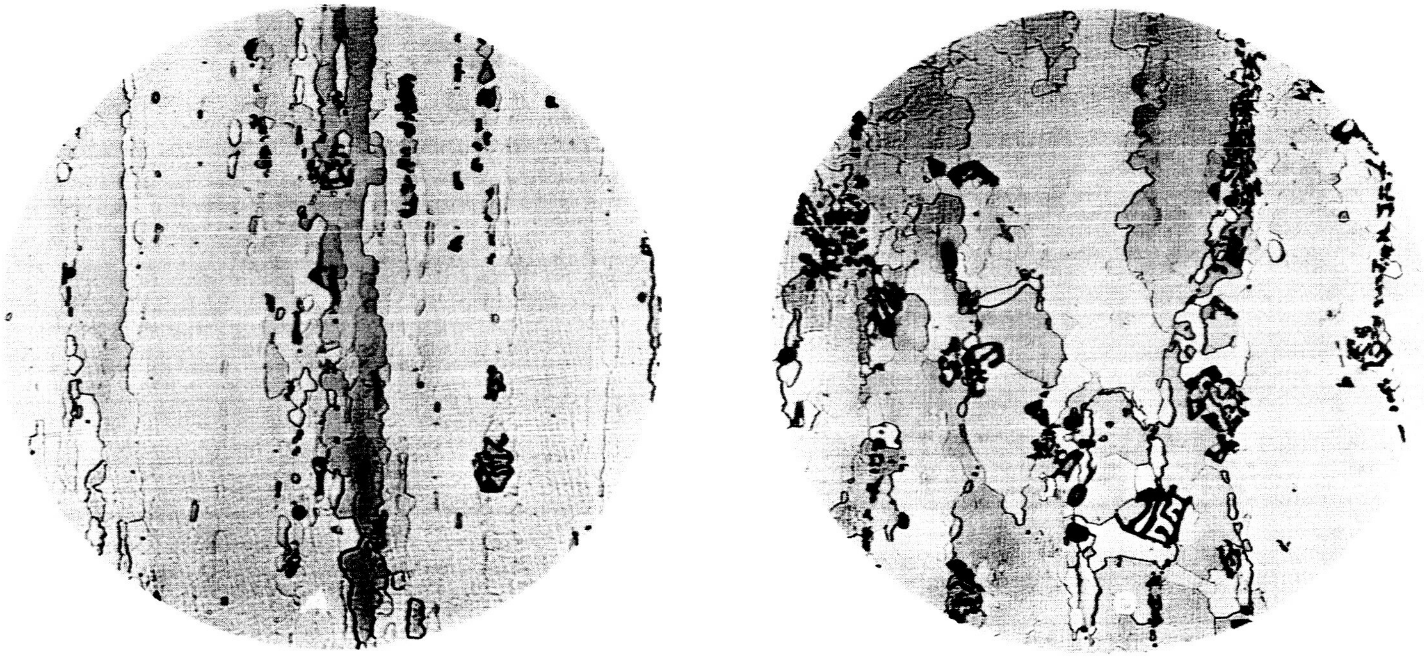


<u>Saturn Helium Bottle</u>

ML 11808C Etchant: Keller's

Photomicrograph of 2014-T651 Aluminum Alloy Extrusion, S/N 0035, showing typical metallographic structure of material in as received condition.

A-Longitudinal Plane
B-Long Transverse Plane
Magn. 400X

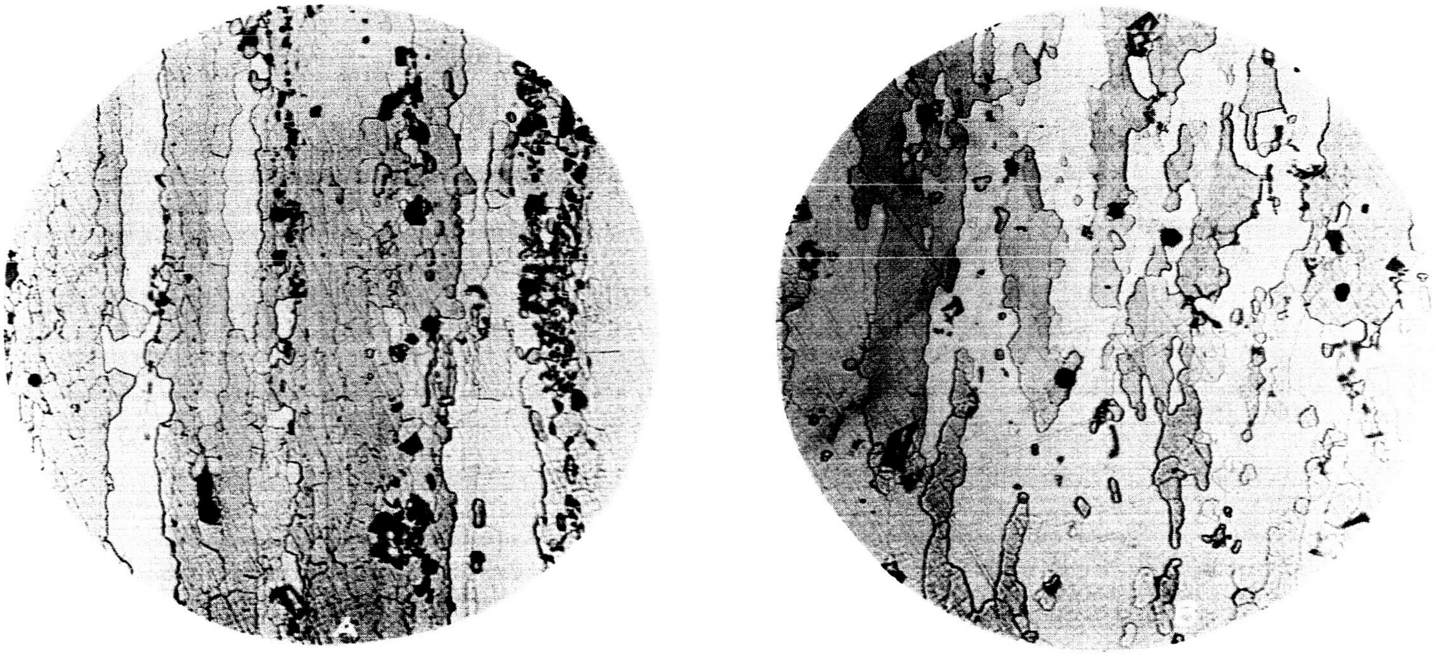


Saturn Helium Bottle

ML 11810C
Etchant:
Keller's

Photomicrograph of 2014-T6 Aluminum Alloy Extrusion, S/N 0035, showing satisfactory metallographic structure of material after solution heat treatment at 945°F/30 minutes soak, cold water quench, and aged at 320°F for 20 hours.

A-Longitudinal Plane
B-Long Transverse Plane
Magn. 400X



Saturn Helium Bottle

ML 11812C
Etchant:
Keller's

Photomicrograph of 2014-T6 Aluminum Alloy Extrusion, S/N 0035, showing typical metallographic structure of material after solution heat treatment at 950°F/30 minutes soak, cold water quench, and aged at 320°F for 20 hours.

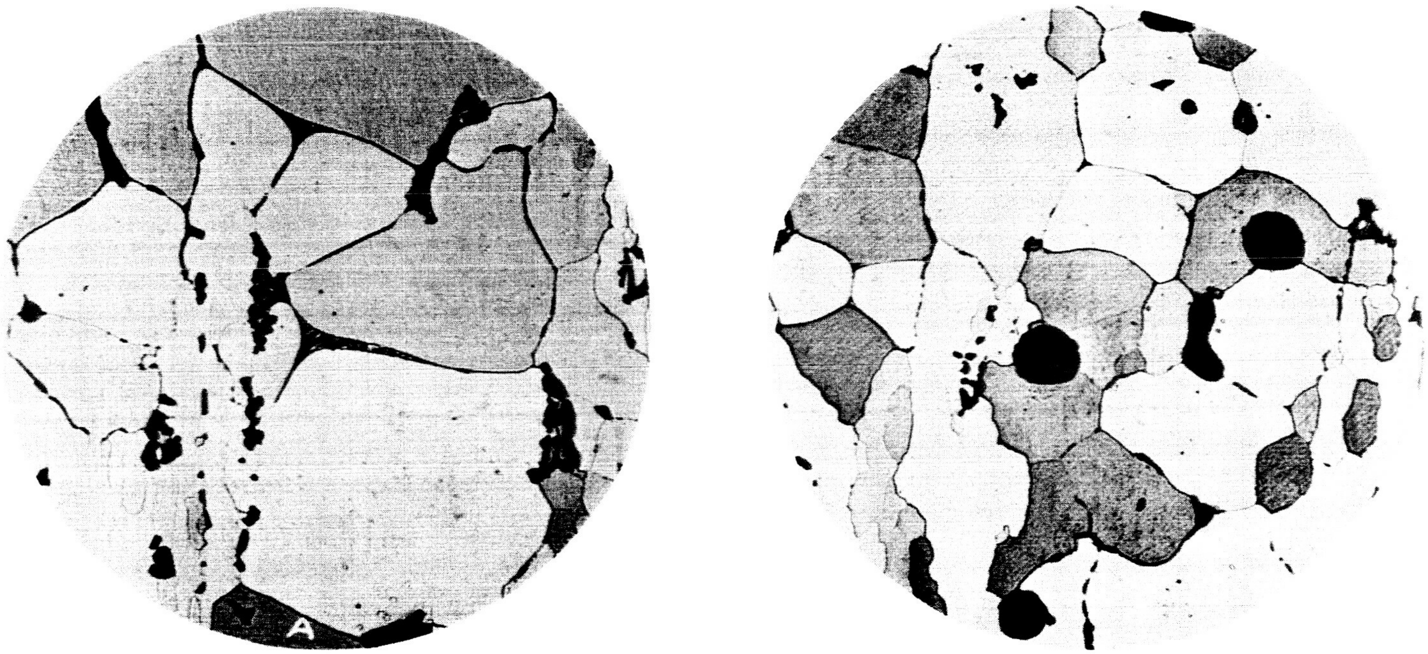
A-Longitudinal Plane
B-Long Transverse Plane
Magn. 400X

Saturn Helium Bottle

ML 11814C
Etchant:
Keller's

Photomicrograph of 2014-T6 Aluminum Alloy Extrusion, S/N 0035, showing typical metallographic structure of material after solution heat treatment at 955°F/30 minutes soak, cold water quench, and aged at 320°F for 20 hours.

A-Longitudinal Plane
B-Long Transverse Plane
Magn. 400X

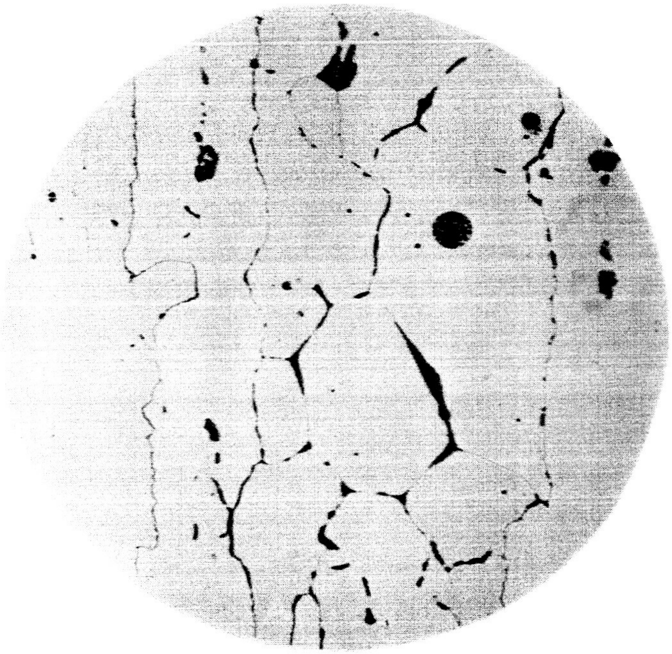


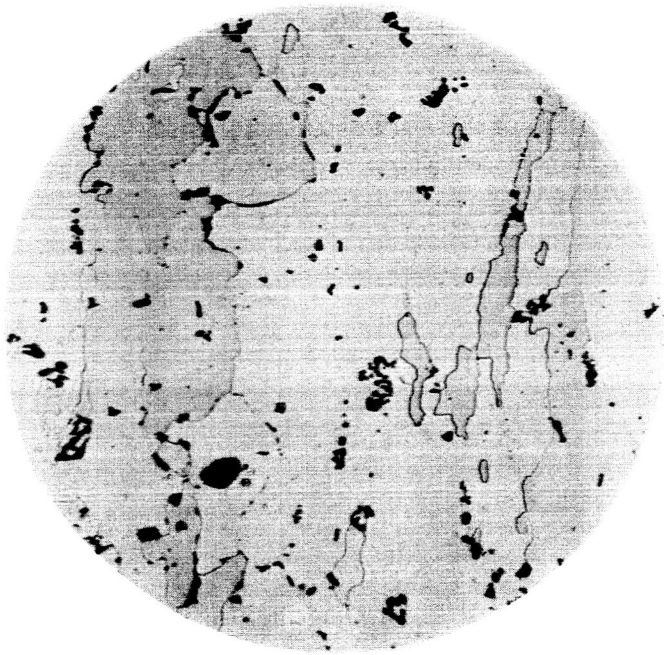
Saturn Helium Bottle

ML 11816C
Etchant:
Keller's

Photomicrograph of 2014-T6 Aluminum Alloy Extrusion, S/N 0035, showing high temperature oxidation, eutectic and solid solution grain boundary melting within metallographic structure after solution heat treatment at 960°F/30 minutes soak, cold water quench, and aged at 320°F for 20 hours.

A-Longitudinal Plane
B-Long Transverse Plane
Magn. 400X



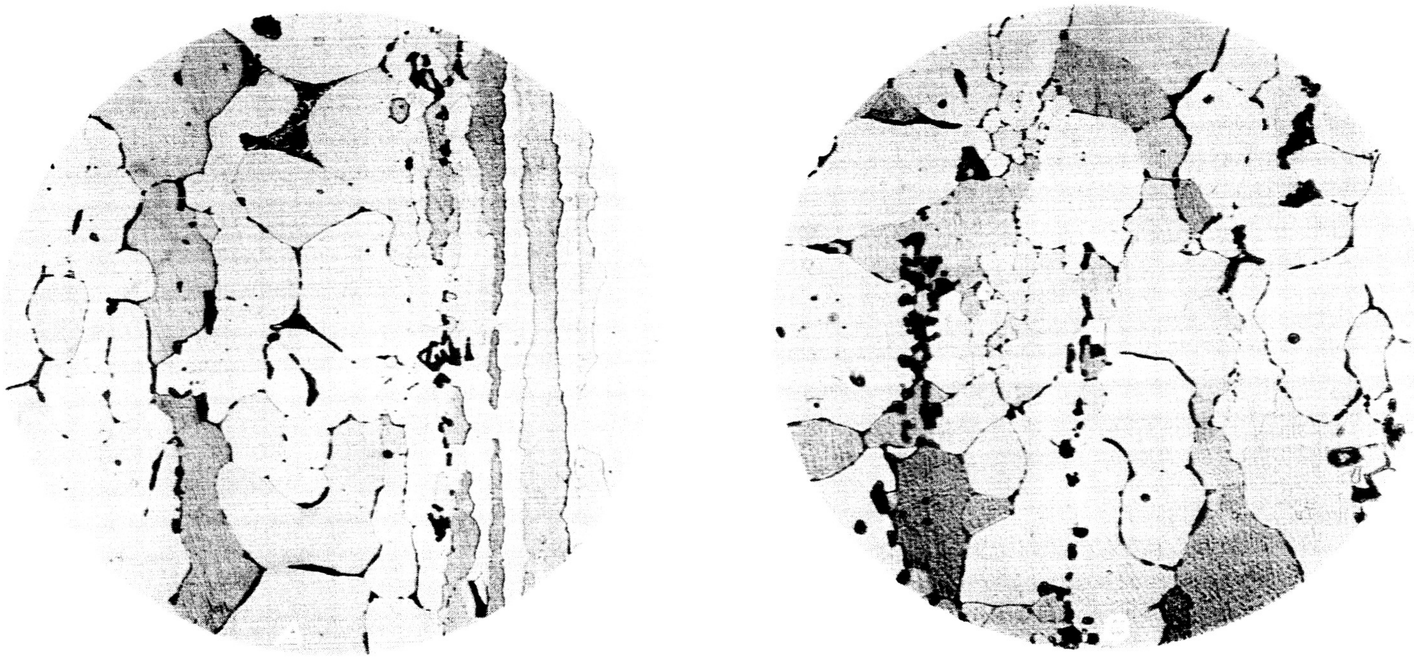


Saturn Helium Bottle

ML 11818C
Etchant:
Keller's

Photomicrograph of 2014-T6 Aluminum Alloy Extrusion, S/N 0035, showing high temperature oxidation, eutectic and solid solution grain boundary melting within metallographic structure after solution heat treatment at 965°F/30 minutes soak, cold water quench, and aged at 320°F for 20 hours.

A-Longitudinal Plane
B-Long Transverse Plane
Magn. 400X



<u>Saturn Helium Bottle</u>

ML 11820C
Etchant:
Keller's

Photomicrograph of 2014-T6 Aluminum Alloy Extrusion, S/N 0035, showing high temperature oxidation, eutectic and solid solution grain boundary melting within metallographic structure after solution heat treatment at 970°F/30 minutes soak, cold water quench, and aged at 320°F for 20 hours.

A-Longitudinal Plane
B-Long Transverse Plane
Magn. 400X